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Chambered circulation evaporator

Background of the Invention

The invention relates to a circulation evaporator for separating mixtures of more volatile and less volatile components, which comprises a discharge container containing at least two chambers. The invention further relates to a process for the separation by distillation of a mixture consisting of a more volatile and a less volatile components.

Circulation evaporators according to the state of the art generally contain an evaporator and a discharge container connected via a connecting piece to said evaporator, a condenser, a vacuum pump, a recirculating line, which connects the discharge container to the evaporator and contains a circulation pump, a feed line for the raw mixture and an outflow line for the purified mixture. The outflow of the purified mixture in this case is generally undertaken from the discharge container; the feed of the raw mixture is generally undertaken into the recirculating line, through which the mixture is pumped from the discharge container to the evaporator. A circulation evaporator according to the state of the art is represented in Figure 1.

Typical examples of circulation evaporators according to the state of the art are described, for example, in Dr Hermann Stage, Fettsäure-Geradeausdestillation und -Fraktionierung unter dem Gesichtswinkel der Wirtschaftlichkeit, CZ-Chemie-Technik, Annual Set 2, pages 254-260 [1973]; Joachim Gebel, Gülle - Aufbereitung: umweltgerecht und wirtschaftlich, WLB, Wasser, Luft und Boden 6, pages 20-26 [1992]; B. Gericke, Zwangsumlaufverdampfer im GUD-Prozeβ und Druckaufgeladenen Systemen, Part 1: Atmosphärischer GUD-Verdampfer, Brennst.-Wärme-Kraft, Vol. 4, No. 6, pages 247-256 [1992] and B. Gericke, Zwangsumlaufverdampfer im GUD-Prozeβ und in druckaufgeladenen Systemen, Part 2: Druckaufgeladener Kohlegas-Verdampfer, Brennst.-Wärme-Kraft, Vol. 44, No. 7/8, pages 295-304 [1992].

If circulation evaporators are used for separating lacquer-resin/isocyanate mixtures, it is necessary to work at quite high temperatures and at low absolute pressures in order to adjust the desired purity of the lacquer resin. In order to avoid excessive temperatures, the circulation evaporators have to make a large evaporation surface available. This results in large items of apparatus and long dwell-times.

In the case of excessive temperatures and excessive residence-times under temperature loading a partial decomposition of the lacquer resin can occur, as well as a change in the color of the lacquer resin, manifesting itself in an increase in the color index.

It is an object of the present invention to provide a circulation evaporator with which the evaporation temperature can be lowered and the thermal load on the mixture consisting of more volatile and less volatile components can be diminished. It is another object of the invention to provide a process for the separation by distillation of a mixture containing more volatile and less volatile components at reduced evaporation temperature.

SUMMARY OF THE INVENTION

The invention relates to a circulation evaporator containing

an evaporator and a discharge container connected via a connecting piece to said evaporator,

a condenser,

a vacuum pump,

a recirculating line, which connects the discharge container to the evaporator and contains a pump, a feed line for a raw mixture,

and an outflow line for the purified mixture,

wherein the discharge container contains at least a chamber for the feed and a chamber for the outflow, and

the chamber for the outflow, to which the outflow line for the purified mixture is connected, is directly connected to the connecting piece for the discharge of the unevaporated liquid from the evaporator, and

the chamber for the feed, into which the feed line for the raw mixture leads, is directly connected to the condenser.

The invention will be described in exemplary manner on the basis of the following Figures.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows a circulation evaporator according to the state of the art,

Figure 2 shows a circulation evaporator according to the invention with chambers of the discharge container interconnected in parallel,

Figure 3 shows an alternative embodiment of a discharge container with chambers interconnected in parallel,

Figure 4 shows an alternative embodiment of a discharge container with chambers interconnected in parallel,

Figure 5 shows a circulation evaporator with serially interconnected chambers of the discharge container.

DETAILED DESCRIPTION OF THE INVENTION

The invention relates to a circulation evaporator containing an evaporator and a discharge container connected via a connecting piece to said evaporator, a condenser, a vacuum pump, a recirculating line, which connects the discharge container to the evaporator and contains a pump, a feed line for the raw mixture and an outflow line for the purified mixture, characterized in that the discharge container consists at least of a chamber for the feed and of a chamber for the outflow, the chamber for the outflow, to which the outflow line for the purified mixture is connected, being directly

connected to the connecting piece for the discharge of the unevaporated liquid from the evaporator, and the chamber for the feed, into which the feed line for the raw mixture leads, being directly connected to the condenser.

In one possible embodiment the two chambers of the discharge container are connected in parallel and are both connected to the evaporator via the connecting piece and both comprise a connection to the pipeline.

In an alternative embodiment the two chambers of the discharge container are serially interconnected, the chamber for the feed, into which the feed line for the raw mixture leads, being arranged downstream of the chamber for the outflow, from which the outflow line for the purified mixture leads, and comprising the connection for the pipeline, and the chamber for the outflow being connected to the evaporator via the connecting piece.

The invention also relates to a continuous process for the separation by distillation of a mixture containing a more volatile and a less volatile components, wherein the chambered circulation evaporator according to the invention is employed with chambers of the discharge container connected in parallel, wherein the raw mixture is supplied to the chamber for the feed of the discharge container through the feed line, in said chamber 1 to 95 %, preferably 10 to 80 %, of the more volatile component are evaporated and recondensed in the condenser and ejected from the condenser, the unevaporated portion from the discharge container is charged to the evaporator via a pipeline, in said evaporator 10 to 99 %, preferably 50 to 90 %, of the more volatile component are evaporated and recondensed in the condenser and ejected from the condenser, the unevaporated portion is charged to the two chambers of the discharge container, and a portion of the purified mixture is withdrawn from the chamber for the outflow of the discharge container via the outflow line.

The invention also relates to a continuous process for the separation by distillation of a mixture containing a more volatile and a less volatile component, wherein a circulation evaporator is employed with serially interconnected chambers of the discharge container, wherein the raw

mixture is supplied to the chamber for the feed of the discharge container through the feed line, in said chamber 1 to 95 %, preferably 10 to 80 %, of the more volatile component are evaporated and recondensed in the condenser and ejected from the condenser, the unevaporated portion is charged to the evaporator via a pipeline, in said evaporator 10 to 99 %, preferably 50 to 90 %, of the more volatile component are evaporated and recondensed in the condenser and ejected from the condenser, the unevaporated portion is charged to the chamber for the outflow of the discharge container, from which a portion of the purified mixture is withdrawn via the outflow line, and the remainder of the purified mixture is charged into the chamber for the feed of the discharge container.

The portion of the purified mixture that is withdrawn via the outflow line from the chamber for the outflow of the discharge container is - in continuous, steady-state operation - equal to the difference formed from the mass flux of raw mixture supplied through the feed line and the mass flux condensed in the condenser and ejected.

In one embodiment of the process the raw mixture is preheated prior to entering the discharge container, so that a relatively large quantity of the more volatile component is evaporated in the chamber for the feed.

In another embodiment of the process the circulation evaporator is operated in such a way that the raw mixture flows into the chamber for the feed of the discharge container below the level of the liquid, so that the quantity of raw mixture that has flown in is rapidly heated up as a result of mixing with the purified mixture and the more volatile component is able to evaporate. In this connection, the closer the location of the feed of the raw mixture to the level of the liquid, the greater the proportion of low-boiling component that evaporates in the chamber for the feed of the discharge container, caused by the declining static pressure in the liquid mixture.

Alternatively, the circulation evaporator can also be operated in such a way that, for example, preheated raw mixture flows into the chamber for the feed of the discharge container above the level of the liquid.

The higher the temperature of the raw mixture supplied, the greater the proportion of evaporated low-boiling component in the chamber for the feed of the discharge container.

In one embodiment of the invention, the ratio of the mass flux of mixture circulated through the recirculating line to the mass flux of raw mixture supplied through the feed line amounts to 1 to 100, preferably 3 to 20.

The circulation evaporator is preferably operated at reduced pressure, preferably at an absolute pressure from 1 to 100 mbar, in particularly preferred manner from 2 to 10 mbar.

The process is particularly suitable for the separation of one or more isocyanates, preferably hexamethylene diisocyanate, from lacquer resins or other polyurethanes or mixtures thereof. Mixtures that can be separated with the circulation evaporator according to the state of the art are also suitable for separation in the chambered circulation evaporator according to the invention. Well suited, in particular, are the polyisocyanates with biuret and/or isocyanurate and/or allophanate and/or urethane and/or uretdione structures that are used conventionally for polyurethane lacquer applications or mixed types with the stated structural elements.

Examples

Example 1

Separation by distillation of hexamethylene diisocyanate from a mixture of polyisocyanates with isocyanurate and uretdione structure

A circulation evaporator according to Figure 2 was employed for the distillation. A mass flux of 800 kg/h having the composition 29 wt.% lacquer resin and 71 wt.% hexamethylene diisocyanate with a temperature of 120°C was conducted as feed 7 into the chamber for the feed 4a of the discharge

container 4 and mixes in said chamber with the liquid discharge from the evaporator 1, which discharges into the chamber for the discharge 4b and into the chamber for the feed 4a of the discharge container 4 with a temperature of 150°C. In the process, a portion of the hexamethylene diisocyanate that was introduced with the feed 7 into the chamber for the feed 4a was evaporated.

The mass flux that was evaporated from the evaporator 1 and from the chamber for the feed 4a of the discharge container 4 was conveyed by the vacuum pump 3, which generates an absolute pressure of 10 mbar, into the condenser 2 and was condensed therein. The mass flux that was ejected from the condenser 2 amounts to 530 kg/h and had the composition of 100 wt.% hexamethylene diisocyanate.

The mass flux that was discharged via the outflow line 8 from the chamber for the discharge 4b of the discharge container 4 amounts to 270 kg/h and had the composition of 86 wt.% lacquer resin and 14 wt.% hexamethylene diisocyanate.

The chamber 4b for the discharge and the chamber 4a for the feed were connected to one another in communicating manner.

The pump 6 conveyed a mass flux of about 7000 kg/h at a temperature of 140°C out of the two chambers 4a and 4b of the discharge container 4 to the top of the evaporator 1 through the recirculating line 5.

Example 2

Separation by distillation of hexamethylene diisocyanate from a mixture of polyisocyanates with biuret structure

A circulation evaporator according to Figure 2 was employed for the distillation. A mass flux of 1450 kg/h having the composition 42 wt.% lacquer resin and 58 wt.% hexamethylene diisocyanate with a temperature of 145°C was conducted as feed 7 into the chamber for the feed 4a of the discharge container 4 and mixed in said chamber with the liquid discharge from the

evaporator 1, which discharged into the chamber for the discharge 4b and into the chamber for the feed 4a of the discharge container 4 with a temperature of 165°C. In the process, a portion of the hexamethylene diisocyanate that was introduced with the feed 7 into the chamber for the feed 4a was evaporated.

The mass flux that was evaporated from the evaporator 1 and from the chamber for the feed 4a of the discharge container 4 was conveyed by the vacuum pump 3, which generated an absolute pressure of 10 mbar, into the condenser 2 and was condensed therein. The mass flux that was ejected from the condenser 2 amounts to 800 kg/h and has the composition of 100 wt.% hexamethylene diisocyanate.

The mass flux that was discharged via the outflow line 8 from the chamber for the discharge 4b of the discharge container 4 amounts to 650 kg/h and had the composition of 94 wt.% lacquer resin and 6 wt.% hexamethylene diisocyanate.

The chamber 4b for the discharge and the chamber 4a for the feed were connected to one another in communicating manner.

The pump 6 conveyed a mass flux of about 10,000 kg/h at a temperature of 155°C out of the two chambers 4a and 4b of the discharge container 4 to the top of the evaporator 1 through the recirculating line 5.

Claims

1. A circulation evaporator comprising

an evaporator and a discharge container connected via a connecting piece to said evaporator,

a condenser,

a vacuum pump,

a recirculating line, which connects the discharge container to the evaporator and contains a pump, a feed line for a raw mixture,

and an outflow line for the purified mixture,

wherein the discharge container comprises at least a chamber for the feed and a chamber for the outflow, and

the chamber for the outflow, to which the outflow line for the purified mixture is connected, is directly connected to the connecting piece for the discharge of the unevaporated liquid from the evaporator, and

the chamber for the feed, into which the feed line for the raw mixture leads, is directly connected to the condenser.

2. The circulation evaporator of claim 1 comprising

the evaporator (1) and the discharge container (4) connected via the connecting piece (9) to said evaporator,

the condenser (2),

the vacuum pump (3),

the recirculating line (5), which connects the discharge container (4) to the evaporator (1) and contains the pump (6), the feed line (7),

and the outflow line (8),

wherein the discharge container (4) comprises the chamber for the feed (4a) and the chamber for the outflow (4b), and

the chamber for the outflow (4b), to which the outflow line (8) for the purified mixture is connected, is directly connected to the connecting piece (9) for the discharge of the unevaporated liquid from the evaporator (1), and

the chamber for the feed (4a), into which the feed line (7) for the raw mixture leads, is directly connected to the condenser (2).

3. The circulation evaporator of claim 1 wherein the two chambers (4a; 4b) of the discharge container (4) are connected in parallel, and are both connected to the evaporator via the connecting piece (9) and both comprise a connection to the recirculating line (5).

4. The circulation evaporator of claim 1 wherein the two chambers (4a; 4b) of the discharge container (4) are connected serially, and

the chamber for the feed (4a) is arranged downstream of the chamber for the outflow (4b).

5. A process for the separation by distillation of a mixture comprising a more volatile and a less volatile component in the circulation evaporator of claim 1, comprising

a) supplying a raw mixture to the chamber for the feed of the discharge container through the feed line, evaporating in said chamber 1 to 95 %, based on the raw mixture, of the more volatile component followed by recondensing the more volatile component in the condenser and ejecting the recondensed more volatile component from the condenser, and

b) charging the unevaporated portion of step a) to the evaporator from the discharge container via a pipeline, and evaporating in the evaporator 10 to 99 %, based on the more volatile component of the unevaporated portion, of the more volatile component followed by recondensing in the condenser and ejecting the recondensed more volatile component from the condenser, and

c) charging the unevaporated portion of step b) to the two chambers of the discharge container, and

d) withdrawing a portion of the purified mixture from the chamber for the outflow of the discharge container via the outflow line.

6. The process of claim 5 wherein the two chambers (4a; 4b) of the discharge container (4) of the circulation evaporator are connected in parallel and are both connected to the evaporator via the connecting piece (9) and both comprise a connection to the pipeline (5).

7. The process of claim 5 wherein in chamber (4a) 10 to 80 % of the more volatile component are evaporated.

8. The process of claim 5 wherein in the evaporator (1) 50 to 90 % of the more volatile component are evaporated.

9. The process of claim 5 wherein the two chambers (4a; 4b) of the discharge container (4) of the circulation evaporator are connected serially, and

the chamber for the feed (4a) is arranged downstream of the chamber for the outflow (4b) comprising

c) charging the unevaporated portion of step b) to the chamber for the outflow (4b) of the discharge container (4),

d) withdrawing a portion of the purified mixture from the chamber for the outflow (4b) via the outflow line (8), and

e) charging the remainder of the purified mixture from the chamber for the outflow into the chamber for the feed (4a) of the discharge container (4).

10. The process of claim 5 wherein the raw mixture is preheated prior to entering the discharge container (4).

11. The process of claim 5 wherein the circulation evaporator is operated in such a way that the raw mixture flows into the chamber for the feed (4a) of the discharge container (4) below the level of the liquid.

12. The process of claim 5 wherein the ratio of the mass flux of mixture circulated through the recirculating line (5) to the mass flux of raw mixture supplied through the feed line (7) amounts to 1 to 100.

13. The process of claim 5 wherein the ratio of the mass flux of mixture circulated through the recirculating line (5) to the mass flux of raw mixture supplied through the feed line (7) amounts to 3 to 20.

14. The process of claim 5 wherein at least one isocyanate is separated from at least one polyurethane or polyisocyanate.

15. The process of claim 14 wherein the the isocyanate is hexamethylene diisocyanate.